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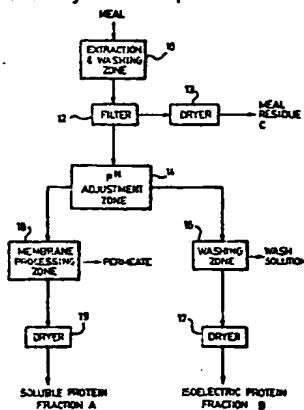
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AT BE CH DE ES FR GB GR IT LI LU NL SE(71) Applicant: The University of Toronto  
Innovations Foundation  
203 College Street, Suite 205  
Toronto Ontario M5T 1P9(CA)(72) Inventor: Diosady Levente L.  
20 York Road  
Willowdale, Ontario M2L 1H5(CA)  
Inventor: Rubin Leon J.  
400 Walmer Road  
2008, Toronto, Ontario M5P 2X7(CA)  
Inventor: Tzeng, Yew-Min  
1040 Woodbine Avenue  
Toronto, Ontario M4C 4C4(CA)(74) Representative: Harding, Richard Patrick et al  
Arthur R. Davies & Co. 27 Imperial Square  
Cheltenham GL50 1RQ(GB)

## (54) Production of rapeseed protein materials.

(57) There is disclosed a process of treating meal containing vegetable proteins. This process includes the step of extracting the meal at an extraction and washing zone 10 with a suitable aqueous solvent, e.g. aqueous sodium hydroxide, in which the vegetable proteins are soluble to obtain an extraction solution. The wet residue is suitably separated, e.g. by filter 12, from the extraction solution, and dried in dryer 13 to provide a meal residue C. The solubility of the dissolved protein in the extraction solution is then adjusted in zone 14 to precipitate at least some of the protein and therefore obtain a precipitated protein fraction and an unprecipitated protein fraction in solution. The precipitated protein fraction is then separated from the protein fraction in solution, and the unprecipitated protein fraction is separated from the undesirable components in the solution by membrane processing in a zone 18. Each of the protein fractions is then suitably dried in dryers 17 and 19 to recover the proteins. This process provides a commercially viable process for producing pure, good-grade protein from rapeseed.



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## Production of Rapeseed Protein Materials

This invention relates to the treatment of meal and other materials containing vegetable protein. More particularly, it relates to methods of extracting vegetable proteins from these materials.

### 5 BACKGROUND OF THE INVENTION

Brassica seeds, especially rapeseed, after oil extraction are a potential source of high-quality protein. After oil extraction, rapeseed meal contains about 38% protein compared to approximately 44% in soybean meal, the latter being widely used for feed and food purposes. Proteins contained in rapeseed are rich in lysine and contain adequate quantities of methionine, both of which are limiting amino acids in most cereal and oilseed proteins. However, the use of rapeseed as a protein source in food products has been severely limited as the proteinaceous material which is left over after oil extraction by known methods contains unwanted constituents such as glucosinolates, phenolics, phytates, and hull, which should be removed from the protein meal of these seeds or at least reduced in quantity therein, in order for the meal and the proteins derived therefrom to be acceptable for human consumption.

The reduction or removal of glucosinolates is particularly important, since they are broken down by enzymes present in the seed and in the human body, producing various degradation products that interfere with thyroid function in the body. Thus for human food use, the glucosinolate content of, for example, proteins derived from rapeseed meal should be substantially eliminated to ensure complete product safety. Phenolic compounds impart a bitter flavour and dark colour to the final protein products. Phytates are strong chelating agents and affect the utilization of polyvalent metal ions, especially zinc and iron, by strongly binding these metals and making them unavailable for metabolism. The hull is present in large amounts and is indigestible for humans and other monogastric animals. It also gives an unsightly heterogeneous product.

These unwanted constituents are difficult to separate from proteins in the rapeseed meal. Unlike other protein-rich oilseeds such as soybean, peanuts and sunflowerseed, rapeseed has a complex protein composition and contains proteins with widely different isoelectric points and molecular weights. Accordingly, the production of isoelectrically precipitated rapeseed-protein isolates with 90% protein content requires complex processes which result in low yields. Also, the products generally contain high concentrations of phytates, present as protein-phytate complexes. Thus, traditional protein-isolation processes are economically and technically unattractive for the production of high-quality rapeseed proteins.

At present, there seem to be no rapeseed-protein isolates in commercial production. In a recent review article prepared for the Canola Council of Canada, Youngs (see "Technical Status Assessment of Food Proteins from Canola", Canola Council of Canada, October 1985) concluded that, in spite of extensive research, the presence of glucosinolates, phytates, phenolics, and hull still represents a serious problem.

In general, protein isolates from rapeseed material have been extracted experimentally, using multi-solvent co-current or counter-current operations. The extracted proteins are recovered in these operations by precipitation at one or more isoelectric points, sometimes enhanced by heat coagulation or complex formation followed by washing and drying. These experimental processes are too complicated and expensive to be used as viable commercial processes.

Membrane processing by ultrafiltration and/or diafiltration has become a popular laboratory technology for vegetable protein isolation in recent years. Ultrafiltration is a technique for separating dissolved molecules on the basis of their size, shape and flexibility by passing their solution through a membrane which acts as a filter with pore diameters suitable for retaining large molecules. Diafiltration is a special technique of ultrafiltration for the removal of small-molecular-weight compounds from an aqueous solution also containing large molecules.

U.S. Patent 4,420,425, which issued to Lawhon on December 13, 1983, explored the potential applications of solubilization and ultrafiltration in soybean and peanut protein systems. Protein extraction of rapeseed was not considered.

In attempting to break the protein-phytate complex to remove the phytate associated with the processing of soybean, peanut, cottonseed and the like, U.S. Patent No. 3,736,147 dated May 29, 1973, which issued to Iacobucci et al proposed to use diafiltration with 0.2 molar calcium chloride solution at a pH of 3, or phytase in the pH range of 4.5 - 7. The process of this patent is directed towards preparing protein products from soybean, cottonseed, peanut, and sesame seed. Protein extraction of rapeseed is not

considered.

A rapeseed-protein isolate has been reported by Von Bockelmann et al in "Potential Applications in Food Processing", in the chapter "Reverse Osmosis and Synthetic Membranes", Ed. S. Sourirajan, page 445, National Research Council of Canada, Publication No. NRCC15627, 1977. This isolate contained 30% protein. The isolate produced by Diosady et al (J. Food Sci. 44:768, 1984) contained 80% protein, but was high in phytate.

A rapeseed protein product with a 76% protein content was obtained by Maubois et al using ultrafiltration in U.S. Patent No. 3,993,636 which issued November 23, 1976. However, a protein content of 76% is too low to be considered an isolate, and since the extraction of the meal was carried out at pH 9, where rapeseed protein solubility is not very high, the protein yield was likely low, although unreported.

Although aqueous sodium hydroxide solutions are effective solvents for rapeseed protein extraction and give high extraction yields, the isoelectric precipitation of these extracts results in low yields, low protein content in the isolates, or both. Likewise, current applications of ultrafiltration and/or diafiltration to rapeseed protein extraction do not result in high-quality rapeseed protein isolates. Thus the problem of developing a commercially viable process for producing pure, food-grade protein from rapeseed up to the present remains unsolved.

## SUMMARY OF THE INVENTION

The present invention provides a process of treating meal containing vegetable protein which comprises:

extracting the meal with a suitable aqueous solvent in which the vegetable proteins are soluble to obtain an extraction solution,

adjusting the solubility of the dissolved proteins in this extraction solution to precipitate at least some of the proteins and obtain a precipitated protein fraction and an unprecipitated protein fraction in solution,

separating the precipitated protein fraction from the unprecipitated protein fraction,

and separating the unprecipitated protein fraction from the undesirable components in the solution by membrane processing.

Preferably, the solvent used in extracting the meal is an alkaline solution. Preferably, the solubility of the dissolved protein in the extraction solution is adjusted by adjusting the pH of the solution. It is also preferred that the membrane processing is by ultrafiltration and/or diafiltration.

Unwanted constituents are extracted and separated from the pure protein fractions in the meal of oil-bearing seeds by adjusting the solubility of the meal solution to separate the insoluble phytates, hull, and meal residue from the extraction solution containing soluble proteins. The solubility of the dissolved protein in the extraction solution is then adjusted so as to separate the insoluble protein fraction and leave the soluble protein fraction in solution. Membrane processing techniques are subsequently used to remove additional unwanted, low-molecular-weight constituents from the soluble protein fraction, and to concentrate and isolate this protein fraction.

By means of the process of the present invention, there can be obtained from the meal of various oil-bearing seeds, high-quality protein fractions that are low in unwanted constituents such as phytates, glucosinolates, phenolics, and hull and are aesthetically and nutritionally appealing. Typically, 90% or more of the recoverable protein originally present in the seeds may be recovered. Each of the protein isolates resulting from the process of the present invention has its own specific functional properties. Thus they may serve as versatile ingredients in food formulations. Moreover, a residual meal with a moderate protein concentration of between 30-40% and substantially free of glucosinolates may be produced which is a good feed-grade material.

## BRIEF REFERENCE TO THE DRAWINGS

The invention will now be described by way of illustration only, with respect to the following drawing  
 5 which is a diagrammatic process flow sheet of a specific preferred process according to the invention.

## DESCRIPTION OF THE BEST MODE

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Referring to the drawing, the starting meal, substantially free from oil, is extracted at an extraction and washing zone 10 with an aqueous alkaline solution e.g. aqueous sodium hydroxide, to dissolve the protein component into the extraction solution and leave most of the hull and phytate undissolved in a wet residue. This wet residue is suitably separated e.g. by filter 12 from the extraction solution, and dried in dryer 13 to  
 15 provide a meal residue C. The pH of the resultant extraction solution is subsequently adjusted in zone 14 with an acid solution to alter the solubility of its component proteins and produce a protein-precipitate fraction and a protein-solution fraction. The two protein fractions are suitably separated. The protein-precipitate fraction is washed in a washing zone 16 with acidic water and dried in dryer 17 to provide an isoelectric-protein fraction B. The protein-solution fraction is subjected at membrane processing zone 18 to  
 20 ultrafiltration followed by diafiltration and is then dried in dryer 19 to provide a soluble protein fraction A.

In a preferred form, the pH of the extraction solution is between 10 and 12.5 at which the proteins are dissolved and most of the phytates and hull undissolved. Variations in this pH value will effect the yields of the protein isolates and the solubility of the phytates. Suitably, the extraction solution is an aqueous solution of an alkaline compound such as sodium hydroxide, potassium hydroxide, or calcium hydroxide.

25 It is desired that the solvent-to-meal ratio for the extraction be between 8 and 50, most preferably 18, followed by washing with solvent to meal ration of 5 to 20.

Due to the low solubility of the phytates and fibre in the preferred pH range, they may be readily separated by conventional techniques from the extraction solution in the form of a wet residual meal, which may be suitable, when dried, for use as an animal feed.

30 The solubility of the dissolved proteins in the resultant extraction solution is adjusted to precipitate one fraction of the protein, the insoluble or isoelectrically precipitated protein fraction, from the other fraction, the soluble protein fraction in solution. Suitably, the solubility is adjusted by adjusting the pH by adding any aqueous acid solution such as hydrochloric, sulphuric, phosphoric or acetic acid, which separates the insoluble and soluble protein fractions. Preferably, the addition of the aqueous acid solution lowers the pH  
 35 to between 2-8, most preferably 3.5 for rapeseed or canola.

To further reduce the phytate level in the soluble protein fraction, up to 50% by weight of the starting meal of calcium chloride may optionally be added.

The two protein fractions, the insoluble protein fraction as a precipitate and the soluble protein fraction in solution, may be appropriately separated from one another by conventional separation techniques.

40 The precipitated insoluble protein fraction is subsequently washed with a suitable solution to purify it further. It is preferred that the washing solution be acidified water at the same pH as that of the previous solution from which the protein was separated, and that the solvent-to-precipitate ratio be between 5-20, most preferably 10. The protein precipitate may be repeatedly washed with such a solution if the concentration of the impurities is high, as long as the protein loss is not too great. The result, after a  
 45 suitable drying, is a high-quality, isoelectrically precipitated protein.

The soluble protein fraction in solution is concentrated and separated from of the low-molecular-weight, potentially toxic impurities such as glucosinolates and the hydrolysis products thereof by a membrane process. Most preferably, this membrane process comprises an ultrafiltration step to concentrate the protein fraction, followed by a diafiltration step to further purify the soluble protein. The permeate, after the  
 50 diafiltration step, in which there is minimal protein loss, is discarded. The resulting retentate is dried to produce a high-quality soluble protein. Filtration membranes of 5-50 kilodaltons molecular weight cut-off provide good protein recovery and impurity removal. For rapeseed protein processing, a 10 kilodalton membrane is preferred.

Based on the concentration of the protein solution to undergo membrane processing and the purity of  
 55 the product required, the concentration factor (CF) of ultrafiltration operation may be varied from 5-20, and the diavolume (DV) of diafiltration may be varied from 3-15. Most preferably, a CF of 10 and DV of 5 were used to optimally reduce the concentration of permeable impurities by up to three orders of magnitude.

The steps prior to membrane processing had separated, by way of solubility differences, the dissolved

protein into two fractions, so that the concentration of protein remaining in solution was relatively low. Thus, the subsequent processing techniques proceeded more efficiently with the elimination of the usual gelation and plugging problems associated with membrane processing in protein production systems.

Optionally, antioxidants such as sodium sulphite may be added during extraction to improve the colour of the final products.

Any type of oil-free meal containing protein can suitably be processed by the process of the present invention. Most suitably processed are oilseeds derived from Brassica seeds especially rapeseed and canola, which are essentially free from oil components, and ground if necessary, before alkaline extraction and washing to allow the subsequent separation of the soluble protein fraction from the insoluble phytate and hull.

An oil-extraction method developed by the inventors for the treatment of rapeseed simultaneously with methanol-ammonia and hexane is suitable for processing the oilseeds prior to their treatment by the process of the present invention. This method is described in U.S. Patent No. 4,469,504 which issued on July 17, 1984 to Rubin et al, Can. Inst. Food Sci. Technol. J. 19. 57, 1986, and Diosady et al, Can. Inst. Food Sci. Technol. J. 18, 121, 1985, the contents of all of which are incorporated herein by reference.

Whilst the process of the invention shows great advantages when applied to commercially prepared meals such as canola and rapeseed meals, and other Brassica meals and flours, it is, as described hereinabove especially suitable to meals from novel oil extraction techniques such as hexane-extracted and air-dried canola meal, methanol/ammonia/hexane-extracted canola meal, dehulled hexane-extracted and air-dried canola meal and dehulled methanol/ammonia/hexane-extracted canola meal. Rapeseed meal is the most preferred starting material. The invention may also be suitable for isolating vegetable proteins from soy, sunflower, peanuts, and cotton seed.

#### Example 1

One hundred grams of methanol/ammonia/hexane-defatted canola meal prepared according to the process of Rubin et al, U.S. Patent No. 4,460,504; 1984 and Rubin et al., "The Alkanol-Ammonia-Water/Hexane Treatment of Canola", Can. Inst. Food Sci. Technol. J. 19: 57, 1986) was stirred for 2 hours with 1800 g water. The pH was kept constant at 12.0 by adding 50% (w/w) NaOH. The slurry was separated by centrifugation (4,080 g, 10 min., 5°C), and the filtrate polished by vacuum filtration using Whatman No. 41 paper. Three batches of the extraction solution were prepared. Two thousand four hundred grams of the extraction solution was acidified to pH 3.5 by adding 6N HCl solution within 4 hours from the time that the meal was first immersed in the alkaline solution. In two of the batches, 5.26 g and 39.26 g of CaCl<sub>2</sub> were added prior to pH adjustment resulting in final CaCl<sub>2</sub> concentrations of 0.02 M and 0.15 M, respectively. After separating the protein precipitate by centrifugation, the solution was filtered. Fifty grams of the wet precipitate was washed with 500 g of pH 3.5 water, and then the washed protein was freeze-dried. In the meantime, 1600 g of the protein solution was concentrated to 160 g by ultrafiltration. One hundred grams of the retentate was diafiltrated with 500 g water. After 500 g of the permeate was collected, the diafiltration retentate was recovered and freeze-dried to produce a water-soluble protein isolate.

The dry-matter content (total solids) of each processing stream was determined. The wet samples were dried at 105°C in a forced-air oven for 24 hours. All freeze-dried samples were oven dried at 105°C overnight prior to analysis. Crude protein (N x 6.25) was determined by the Kjeldahl method. Glucosinolate content was determined by the method of Wetter and Youngs ("A thiourea-UV assay for total glucosinolate content in rapeseed meals", JAOCS, 53:162, 1976). The content of glucosinolate in the meal is expressed as  $\mu$ M equivalent of 3-butenyl-isothiocyanate per gram of sample. Phytate was determined by a procedure adapted from the methods given by Wheeler and Ferrel ("Phytic acid: I. Determination of three forms of phosphorus in flour, dough, and bread", Cereal Chem. 58:226, 1981), as previously described (Naczek et al., "The phytate and complex phenol content of meals produced by alkanol-ammonia/hexane extraction of canola", Lebensm.-Wiss. U.-Technol. 19:3, 1986).

The results, compositions and yields, are given in Table 1.

Most of the nitrogen ( $\approx$  90%) in the starting meal was recovered as three usable products by the process. Approximately 45% of the nitrogen was recovered as protein isolates.

As the concentration of CaCl<sub>2</sub> is increased, the yield ratio of the isoelectric protein to the soluble protein is decreased, due to the "salting in" effect. All protein isolates had protein contents of  $\approx$  90%. To obtain a phytate-free protein isolate, substantial amounts of CaCl<sub>2</sub> must be used. The residual meal had protein content of 33.1% and was free of glucosinolates, which makes it a usable feed material.

Example 2

The procedure of Example 1 was essentially repeated, using, a hexane-extracted and air-dried canola meal as the starting meal in place of the methanol/ammonia/hexane meal. The meal was extracted at pH 11.0 for 30 min. Only one batch of extraction solution was prepared.  $\text{CaCl}_2$  was not used. The pH of the extraction solution was brought down to 3.5 within 2 hours from the moment when the meal was first immersed in the alkaline solution. Total solids, crude protein, glucosinolate, and phytate determinations were carried out as in Example 1. The results are given in Table 2.

The results indicate that 75.4% of the nitrogen in the starting meal was recovered in the isolates. Because approximately 10% nitrogen is bound in the hulls and another 10% is non-protein nitrogen, the recovery of the usable protein was as high as 94.25% ( $75.4/80 \times 100\%$ ). Both isolates were free of glucosinolates and low in phytate.

15

Example 3

The procedure of Example 2 was essentially repeated, except that a dehulled hexane-extracted and air-dried canola meal prepared according to the process of Schneider, Canadian Patent No. 1,062,118; 1979, was used as the starting meal in place of the "whole" meal. The results are given in Table 3.

Only 50% of the nitrogen was recovered as isolates, due to the low solubility observed during extraction. This may be due to the treatment in the dehulling process. However, the residual meal (dehulled) is a high-quality feed material.

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Example 4

The procedure of Example 1 was repeated, with the following changes: a dehulled methanol/ammonia/hexane-extracted canola meal was used as the starting meal; a single extraction batch was used for membrane processes, and no  $\text{CaCl}_2$  was added.

The results are given in Table 4.

The protein solubility was low, due to the treatment in the dehulling process (Schneider, Canadian Patent No. 1,062,118, 1979) and the effect of methanol/ammonia solution. Approximately 42% nitrogen was recovered as high-quality protein isolates. More than 50% nitrogen was recovered in a dehulled residual meal with a protein content of 38.5%, which is a usable feed material.

40 Example 5

The procedure of Example 2 was essentially repeated, except that a commercial canola meal was used as the starting meal, and the extraction was carried out in the presence of 0.0024  $\text{Na}_2\text{SO}_3$  as an anti-oxidant, equivalent to 1% (w/w) of the meal.

The results are given in Table 5.

Approximately - 62%, of the nitrogen was recovered as a meal. Because of low protein solubility which was due to the heat treatment of the proteins during commercial processing, the yield and protein concentration of the isolates were both low. The colour of the isolates was light due to the effect of sodium sulphite.

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Table 1

Effect of precipitation and ultrafiltration/diafiltration process on methanol/ammonia/hexane-extracted  
 5 canola meal (NaOH extraction at pH 12, 10 kD membrane for UF/DF).

## Compositions And Yields

	Protein N x 6.25	Glucosinolates µM/g	Phytic acid%	Yield (as % of starting meal)			
				Solids	N	Glucosinolates	Phytic acid
Starting meal	50.5	0.6	4.32	100	100	100	100
Meal residue	33.1	N/D*	5.63	69.1	45.2	≈ 0	90
<u>1. without CaCl<sub>2</sub> addition</u>							
Isoelectric protein	98.7	N/D	1.56	13.2	25.8	≈ 0	4.8
Soluble protein	94.2	N/D	1.41	11.3	21.0	≈ 0	3.7
LOSS				6.4	8.0	≈ 100	1.5
<u>2. CaCl<sub>2</sub> equivalent to 6.7% of meal solids was added</u>							
Isoelectric protein	100.6	N/D	1.54	12.0	23.9	≈ 0	4.3
Soluble protein	103.1	N/D	1.13	10.7	21.9	≈ 0	2.8
LOSS				8.2	9.0	≈ 100	2.9
<u>3. CaCl<sub>2</sub> equivalent to 50% of meal solids was added</u>							
Isoelectric protein	91.1	N/D	0.73	5.5	9.9	≈ 0	0.9
Soluble protein	90.8	N/D	0	19.9	35.8	≈ 0	0
LOSS				5.5	9.1	≈ 100	9.1

\*Not detected

Table 2

Effect of precipitation and ultrafiltration/diafiltration process on hexane-extracted canola meal (NaOH extraction at pH 11, 10kD membrane for UF/DF).

Compositions And Yields							
	Protein N x 6.25	Glucosinolates $\mu\text{M/g}$	Phytic acid %	Yield (as % of starting meal)			
				Solids	N	Glucosinolates	Phytic acid
Starting meal	39.2	15.3	3.92	100	100	100	100
Meal residue	11.4	N/D	6.51	49.9	14.6	$\approx 0$	82.9
Isoelectric protein	87.4	N/D	1.09	19.2	42.8	$\approx 0$	9.5
Soluble protein	96.1	N/D	1.24	13.3	32.6	$\approx 0$	4.1
LOSS				17.6	10.0	$\approx 100$	3.5

Table 3

Effect of precipitation and ultrafiltration/diafiltration process on hexane-extracted dehulled canola meal (NaOH extraction at pH 11, 10kD membrane for UF/DF).

Compositions And Yields							
	Protein N x 6.25	Glucosinolates $\mu\text{M/g}$	Phytic acid %	Yield (as % of starting meal)			
				Solids	N	Glucosinolates	Phytic acid
Starting meal	52.1	20.4	4.44	100	100	100	100
Meal residue	39.5	N/D	7.64	52.1	39.5	$\approx 0$	89.6
Isoelectric protein	97.3	N/D	2.39	7.5	14.0	$\approx 0$	4.0
Soluble protein	102.5	N/D	1.14	18.0	35.4	$\approx 0$	4.6
LOSS				22.4	11.1	$\approx 100$	1.8



Table 4

Effect of precipitation and ultrafiltration/diafiltration process on methanol/ammonia/hexane-extracted dehulled canola meal (NaOH extraction at pH 12, 10kD membrane for UF/DF).

**Compositions And Yields**

	Protein N x 6.25	Glucosinolates µM/g	Phytic acid %	Yield (as % of starting meal)			
				Solids	N	Glucosinolates	Phytic acid
Starting meal	50.7	3.1	4.68	100	100	100	100
Meal residue	38.5	N/D	6.02	69.3	52.6	≈ 0	89.2
Isoelectric protein	97.8	N/D	2.47	10.1	19.5	≈ 0	5.3
Soluble protein	103.6	N/D	1.33	11.1	22.7	≈ 0	3.2
LOSS				9.5	5.2	≈ 100	2.3

Table 5

Effect of precipitation and ultrafiltration/diafiltration process on commercial canola meal  
(NaOH extraction at pH 11, in the presence of 0.0024M Na<sub>2</sub>SO<sub>3</sub> equivalent to  
1% w/w of the meal, 10kD membrane for UF/DF).

## Compositions And Yields

	Protein N x 6.25	Glucosinolates μM/g	Phytic acid %	Yield (as % of starting meal)			
				Solids	N	Glucosinolates	Phytic acid
Starting meal	44.7	9.7	4.14	100	100	100	100
Meal residue	41.6	N/D	5.72	66.6	61.9	≈ 0	92.0
Isoelectric protein	82.6	N/D	2.00	11.9	22.0	≈ 0	5.7
Soluble protein	86.2	N/D	1.71	5.7	11.0	≈ 0	2.3
LOSS				15.8	5.1	≈ 100	0

## Claims

1. A process of treating meal containing vegetable proteins comprising:

extracting said meal with a suitable aqueous solvent in which said vegetable proteins are soluble, to obtain an extraction solution;

adjusting the solubility of the dissolved proteins in said extraction solution to precipitate at least some of said protein, to obtain an unprecipitated protein fraction in solution and a precipitated protein fraction;

separating the precipitated protein fraction from the protein fraction in solution; and

separating the unprecipitated protein fraction from the undesirable components in the solution by membrane processing.

2. The process according to claim 1, wherein the extraction solvent is an aqueous alkaline solution.

3. The process according to claim 1 or 2, wherein the solubility is adjusted by the addition of an aqueous acid solution.

4. The process according to claim 1, 2 or 3, wherein membrane processing comprises ultrafiltration, diafiltration or ultrafiltration followed by diafiltration.

5. The process according to any preceding claim, wherein the meal containing vegetable proteins is selected from the group comprising hexane-extracted and air-dried canola meal, methanol/ammonia/hexane-extracted canola meal, dehulled hexane-extracted and air-dried canola meal, dehulled methanol/ammonia/hexane-extracted canola meal, and commercially prepared rapeseed meal.

6. The process according to any preceding claim, additionally comprising washing with a suitable acidic solution, and drying said precipitated protein fraction.

7. The process according to any preceding claim, wherein said unprecipitated protein fraction in solution is suitably dried and recovered after membrane processing.

8. The process according to any preceding claim, wherein the extraction solvent is selected from the group comprising sodium hydroxide, potassium hydroxide, and calcium hydroxide.

9. The processes according to any preceding claim, wherein the extraction solvent is of a pH between 10 and 12.5, and preferably between 11 and 12.

10. The process according to claim 3 or any one of claims 4 to 9 when appended directly or indirectly to claim 3, wherein said aqueous acid solution is selected from the group comprising hydrochloric acid, sulphuric acid, phosphoric acid or acetic acid.

11. The process according to any preceding claim, wherein the nominal molecular weight cut-off of the membrane is from 5 to 50 kilodaltons, and preferably about 10 kilodaltons.

12. The process according to claim 4 or any one of claims 5 to 11 when appended directly or indirectly to claim 4, wherein said ultrafiltration has a concentration factor from 5 to 20, and preferably about 10.

13. The process according to claim 4 or any one of claims 5 to 12 when appended directly or indirectly to claim 4, where said diafiltration has a diavolume of from 3 to 15, and preferably about 5.

14. The process according to claim 6 or any one of claims 7 to 13 when appended directly or indirectly to claim 6, wherein the ratio of the acidic washing solution to the precipitated protein fraction is from 5 to 20, and preferably about 10.

15. The process according to any preceding claim, wherein the extraction solvent-to-meal ratio is from 8 to 50, and preferably about 18.

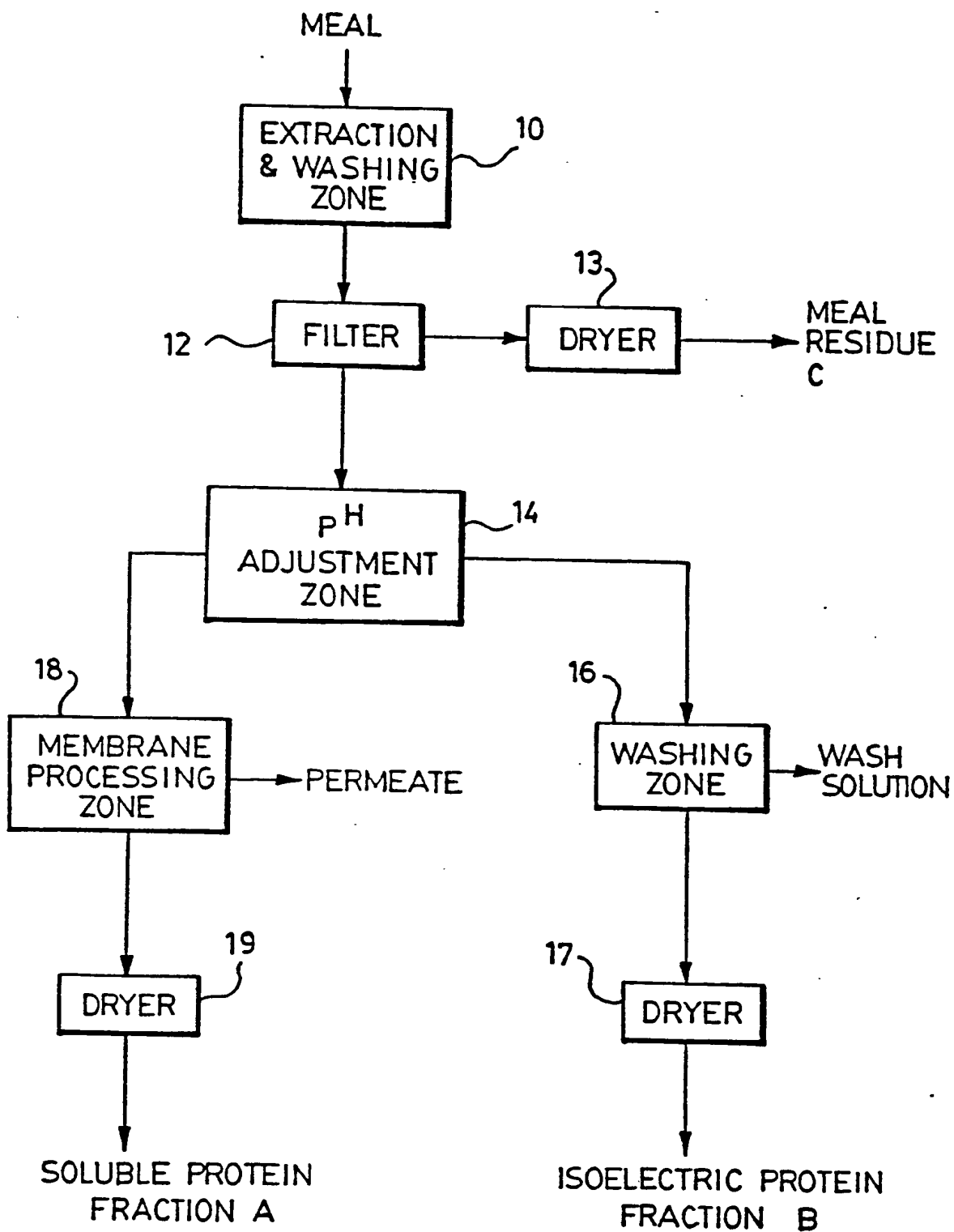
16. The process according to any preceding claim, additionally comprising, during the adjustment in solubility of said extraction solution, the addition of calcium chloride.

17. The process according to any preceding claim, wherein, during said extracting, a suitable antioxidant is added.

18. An isoelectric, precipitated vegetable protein isolate, a soluble vegetable protein isolate or a residual proteinaceous meal when prepared by a process according to any preceding claim.

19. A process of treating meal containing vegetable proteins comprising:

extracting said meal with an aqueous alkaline solution in which said vegetable proteins are highly soluble, to obtain an extraction solution;  
changing the pH of the said extraction solution to between 2 to 8 to precipitate at least some of the said protein and obtain a precipitated protein fraction and an unprecipitated protein fraction in solution;  
recovering said precipitated protein fraction by acidic water washing and drying;  
separating the unprecipitated protein fraction from the undesirable, low-molecular-weight components in the solution by ultrafiltration followed by diafiltration; and  
recovering said unprecipitated protein fraction in solution by drying.





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Applicant: The University of Toronto Innovations  
Foundation  
203 College Street, Suite 205  
Toronto Ontario M5T 1P9 (CA)

Inventor: Diosady Levente L.  
20 York Road  
Willowdale, Ontario M2L 1H5 (CA)

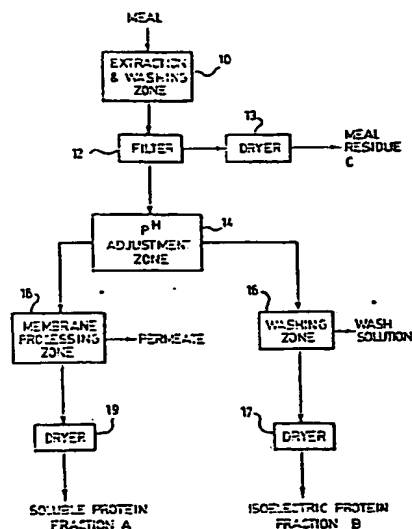
Rubin Leon J.  
400 Walmer Road  
2008, Toronto, Ontario M5P 2X7 (CA)

Tzeng, Yew-Min  
1040 Woodbine Avenue  
Toronto, Ontario M4C 4C4 (CA)

Representative: Harding, Richard Patrick et al  
Arthur R. Davies & Co. 27 Imperial Square  
Cheltenham GL50 1RQ (GB)

### Production of rapeseed protein materials.

There is disclosed a process of treating meal containing vegetable proteins. This process includes the step of extracting the meal at an extraction and washing zone 10 with a suitable aqueous solvent, e.g. aqueous sodium hydroxide, in which the vegetable proteins are soluble to obtain an extraction solution. The wet residue is suitably separated, e.g. by filter 12, from the extraction solution, and dried in dryer 13 to provide a meal residue C. The solubility of the dissolved protein in the extraction solution is then adjusted in zone 14 to precipitate at least some of the protein and therefore obtain a precipitated protein fraction and an unprecipitated protein fraction in solution. The precipitated protein fraction is then separated from the protein fraction in solution, and the unprecipitated protein fraction is separated from the undesirable components in the solution by membrane processing in a zone 16. Each of the protein fractions is then suitably dried in dryers 17 and 19 to recover the proteins. This process provides a commercially viable process for producing pure, good-grade protein from rapeseed.





European Patent  
Office

# EUROPEAN SEARCH REPORT

Application Number

EP 88 30 3455

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. 4)
X	US-A-3 995 071 (K.C. GOODNIGHT) * Claim 1; example 1 *	1-4, 8-11, 15, 18	A 23 J 1/14
Y	---	5	
Y	JOURNAL OF THE AMERICAN OIL CHEMISTS SOCIETY, vol. 58, no. 12, December 1981, pages 1021-1023, Champaign, Illinois, US; R.D. SIY et al.: "Preparation of low-phytate rapeseed protein by ultrafiltration: II. Membrane development and testing" * Page 1021 *	5	
A	---		
A	JOURNAL OF FOOD SCIENCE, vol. 49, no. 3, May/June 1984, pages 768-770, Chicago, Illinois, US; L.L. DIOSDAY et al.: "Preparation of rapeseed protein concentrates and isolates using ultrafiltration" * Page 768, column 2 *	1, 5, 11	
A	---		TECHNICAL FIELDS SEARCHED (Int. Cl. 4)
A	US-A-3 736 147 (G.A. IACOBUCCI et al.) * Example 1 *	1-4	A 23 J
A	---		
A	FOOD TECHNOLOGY, vol. 38, no. 12, December 1984, pages 97-106, Chicago, Illinois, US; J.T. LAWHON et al.: "New techniques in membrane processing of oilseeds" * Page 97, column 1 *	1-4	
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 14-10-1988	Examiner SANTOS Y DIAZ A.I.
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons ----- & : member of the same patent family, corresponding document	

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